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2-Hydroxy-6-[(*m*-tolyliminio)methyl]phenolate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.127
Data-to-parameter ratio = 13.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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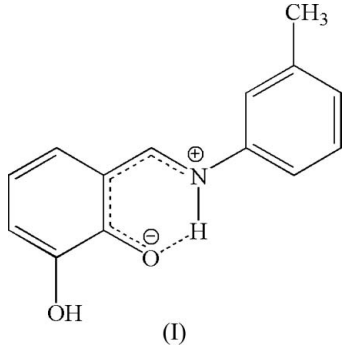
In the solid state, the title compound, $C_{14}H_{13}NO_2$, crystallizes as a zwitterion. Two molecules comprise the asymmetric unit. The molecules exhibit two types of hydrogen bonds: N—H···O hydrogen bonds involving hydroxy and imine groups generate an *S*(6) ring motif, and O—H···O hydrogen bonds linking two symmetry-related molecules form a centrosymmetric dimer.

Comment

Hydroxy Schiff bases have been extensively studied due to their biological, photochromic and thermochromic properties (Garnovskii *et al.*, 1993; Hadjoudis *et al.*, 2004). They can be used as potential materials for optical memory and switch devices (Zhao *et al.*, 2007). Proton transfer in these compounds forms the basis for an explanation of the mechanisms of various biological processes where proton transfer is the rate-determining step (Lussier *et al.*, 1987). The title Schiff base, (I), was obtained by the condensation of 2,3-dihydroxybenzaldehyde with 3-methylaniline.



(I)

The molecular structure of (I) is similar to that of the recently published structure, 2-{(4-carboxycyclohexyl)-methylaminomethyl}-6-hydroxyphenolate (Shuja *et al.*, 2006), which also crystallizes as a zwitterion. The aromatic rings C1–C6 and C8–C13 form dihedral angles of 6.01 (9) and 12.92 (9)° in molecules (I) and (II), respectively. (In the Figures and tables, the first digit of the atom label gives the residue number, *i.e.* molecule 1 or molecule 2). The C8—N1—C7—C6 torsion angles are −177.42 (17) and −174.99 (16)°, respectively. The central C7=N1 bond lengths of 1.301 (2) and 1.305 (2) Å are smaller than the N1—C8 bond lengths of 1.421 (2) and 1.420 (2) Å. The C1—O1 and C6—C7 bond lengths indicate double-bond character.

Molecules 1 and 2 exhibit two types of hydrogen bonds, an intramolecular N—H···O hydrogen bond and two intermolecular O—H···O hydrogen bonds (Fig. 3 and Table 1). In

the crystal packing of (I), symmetry-related molecules are linked by O—H···O hydrogen bonds forming centrosymmetric dimers, which stack along the *b* axis direction.

Also observed are a weak intramolecular O—H···O hydrogen bond and a weak intermolecular C—H···O($x-2$, $1+y$, z) hydrogen bond between molecules 1 and 2 (Table 1).

Experimental

An ethanolic solution (20 ml) of 3-methylaniline (2.5 mmol, 0.27 g) was added dropwise with constant stirring to a hot ethanolic (20 ml) solution of 2,3-dihydroxybenzaldehyde (2.5 mmol, 0.34 g) in a round-bottomed flask equipped with a water condenser. The reaction mixture was kept under reflux for 3 h, cooled and kept at room temperature for 72 h. Red crystals of (I) were obtained on slow evaporation of the solvent.

Crystal data

$C_{14}H_{13}NO_2$
$M_r = 227.25$
Triclinic, $P\overline{1}$
$a = 7.7094$ (7) Å
$b = 10.5939$ (9) Å
$c = 14.8307$ (13) Å
$\alpha = 72.2740$ (15)°
$\beta = 83.0670$ (16)°
$\gamma = 74.2580$ (15)°
$V = 1109.52$ (17) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 100$ (1) K
0.39 × 0.17 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2006)
$T_{min} = 0.955$, $T_{max} = 0.989$
7014 measured reflections
4253 independent reflections
2951 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.127$
$S = 0.99$
4253 reflections
315 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H11···O11	1.00 (2)	1.61 (2)	2.528 (2)	150.8 (18)
O12—H12···O11	0.91 (2)	2.29 (2)	2.719 (2)	108.7 (18)
O12—H12···O11^{i}	0.91 (2)	1.94 (2)	2.774 (2)	152 (2)
N2—H21···O21	0.87	1.82	2.550 (2)	140
O22—H22···O21	0.83	2.30	2.7501 (19)	114
O22—H22···O21^{ii}	0.83	1.97	2.6965 (19)	145
C13—H13···O22^{iii}	0.95	2.57	3.507 (3)	171

Symmetry codes: (i) $-x-1, -y+1, -z+1$; (ii) $-x+2, -y, -z$; (iii) $x-2, y+1, z$.

N- and O-bound H atoms were located in a difference Fourier map and refined with isotropic displacement parameters. All other H atoms were generated by geometrical considerations and were included in the riding-model approximation, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.5$ for methyl and $x = 1.2$ for the other H atoms. The methyl groups were refined as rigid groups, which were allowed to rotate freely. Assigned values of bond distances: methyl C—H = 0.98 Å and aromatic C—H = 0.95 Å. The N—H bonds are involved in an intramolecular hydrogen bond; the observed N—H bond distances are elongated compared with commonly observed values of around 0.85–0.90 Å.

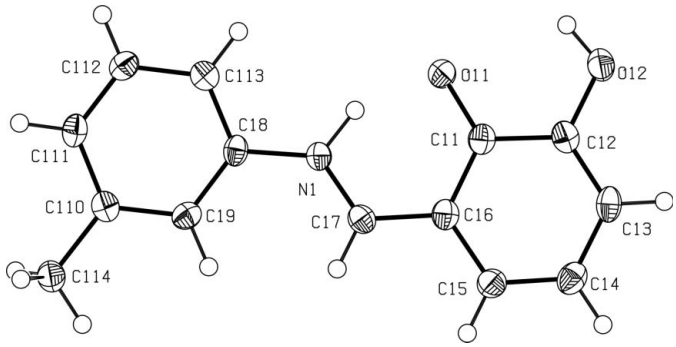


Figure 1
The structure of molecule 1 of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown with an arbitrary radius.

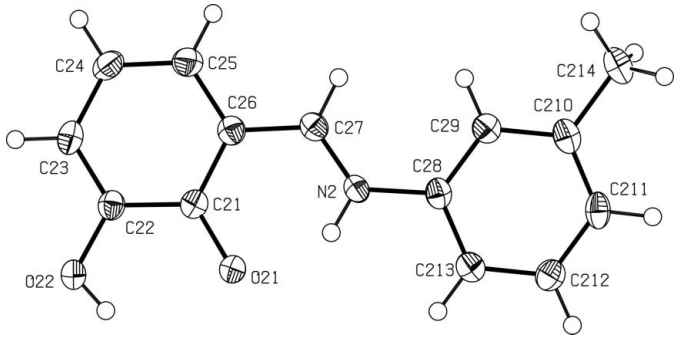


Figure 2
The structure of molecule 2 of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown with an arbitrary radius.

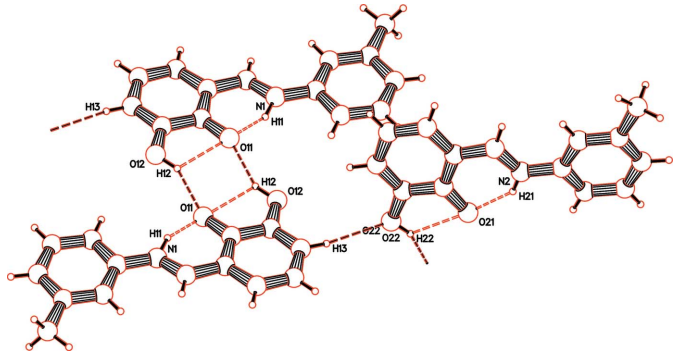


Figure 3
A perspective *PLUTO* (Meetsma, 2007) drawing, showing the dimers formed by O—H···O hydrogen bonds (symmetry code: $-x-1, -y+1, -z+1$) and the aggregates formed by the weak C—H···O bonds (symmetry code: $x-2, y+1, z$). Hydrogen bonds are shown as dashed lines.

Data collection: *SMART* (Bruker, 2006); cell refinement: *SAINT-Plus* (Bruker, 2006); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics:

PLUTO (Meetsma, 2007) and *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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Acta Cryst. (2007). E**63**, o3162–o3164 [https://doi.org/10.1107/S1600536807012561]

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Crystal data

$C_{14}H_{13}NO_2$
$M_r = 227.25$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.7094$ (7) Å
$b = 10.5939$ (9) Å
$c = 14.8307$ (13) Å
$\alpha = 72.2740$ (15)°
$\beta = 83.0670$ (16)°
$\gamma = 74.2580$ (15)°
$V = 1109.52$ (17) Å^3
$Z = 4$
$F(000) = 480$

The final unit cell was obtained from the xyz centroids of 2168 reflections after integration using the SAINT-Plus software package (Bruker, 2000).
Reduced cell calculations did not indicate any higher metric lattice symmetry and examination of the final atomic coordinates of the structure did not yield extra symmetry elements (Spek, 1988; Le Page 1987, 1988)
$D_x = 1.360$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2168 reflections
$\theta = 2.8$–$27.3°$
$\mu = 0.09$ mm^{-1}
$T = 100$ K
Block, red
0.39 × 0.17 × 0.12 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine focus sealed Siemens Mo tube
Parallel mounted graphite monochromator
Detector resolution: 4096x4096 / 62x62 (binned 512) pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2006)

$T_{min} = 0.955$, $T_{max} = 0.989$
7014 measured reflections
4253 independent reflections
2951 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.8°$
$h = -9 \rightarrow 9$
$k = -12 \rightarrow 13$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.048$
$wR(F^2) = 0.127$
$S = 0.99$
4253 reflections
315 parameters
0 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0682P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O11	−0.30956 (17)	0.46121 (13)	0.44824 (10)	0.0294 (4)
O12	−0.61344 (18)	0.62343 (14)	0.35839 (11)	0.0319 (5)
N1	0.0301 (2)	0.37546 (15)	0.44598 (11)	0.0229 (5)
C11	−0.2912 (3)	0.54424 (18)	0.36417 (13)	0.0233 (6)
C12	−0.4475 (3)	0.63121 (19)	0.31473 (14)	0.0249 (6)
C13	−0.4334 (3)	0.71750 (19)	0.22577 (14)	0.0262 (6)
C14	−0.2624 (3)	0.72581 (19)	0.18151 (14)	0.0286 (6)
C15	−0.1079 (3)	0.64563 (19)	0.22702 (13)	0.0269 (6)
C16	−0.1193 (3)	0.55269 (18)	0.31780 (13)	0.0234 (6)
C17	0.0402 (3)	0.46546 (19)	0.36446 (13)	0.0233 (6)
C18	0.1736 (3)	0.27812 (18)	0.49996 (13)	0.0220 (6)
C19	0.3529 (3)	0.25659 (19)	0.46668 (13)	0.0236 (6)
C110	0.4880 (3)	0.16019 (19)	0.52310 (13)	0.0233 (6)
C111	0.4389 (3)	0.08514 (19)	0.61330 (14)	0.0250 (6)
C112	0.2597 (3)	0.10638 (19)	0.64576 (14)	0.0255 (6)
C113	0.1267 (3)	0.20312 (19)	0.58927 (13)	0.0244 (6)
C114	0.6833 (3)	0.1366 (2)	0.48869 (14)	0.0276 (6)
H11	−0.100 (3)	0.388 (2)	0.4670 (14)	0.0331*
H12	−0.605 (3)	0.573 (2)	0.4203 (16)	0.0414*
H13	−0.53950	0.77225	0.19351	0.0315*
H14	−0.25413	0.78699	0.12011	0.0343*
H15	0.00682	0.65269	0.19740	0.0323*
H17	0.15512	0.47332	0.33554	0.0279*
H19	0.38328	0.30804	0.40501	0.0283*
H111	0.52913	0.01890	0.65276	0.0301*
H112	0.22822	0.05432	0.70700	0.0305*
H113	0.00417	0.21789	0.61164	0.0292*
H114	0.69231	0.19175	0.42273	0.0414*
H114′	0.73242	0.03956	0.49277	0.0414*
H114″	0.75207	0.16320	0.52821	0.0414*
O21	0.96821 (18)	0.15125 (13)	0.01479 (9)	0.0283 (4)
O22	1.14752 (19)	−0.09574 (13)	0.13003 (9)	0.0307 (4)
N2	0.8077 (2)	0.40206 (15)	−0.00512 (11)	0.0233 (5)
C21	1.0044 (2)	0.14475 (19)	0.10049 (13)	0.0223 (6)
C22	1.0979 (3)	0.01771 (19)	0.16170 (13)	0.0239 (6)
C23	1.1390 (3)	0.0095 (2)	0.25099 (13)	0.0264 (6)

C24	1.0891 (3)	0.1252 (2)	0.28508 (14)	0.0299 (7)
C25	0.9982 (3)	0.2484 (2)	0.22875 (14)	0.0300 (7)
C26	0.9559 (3)	0.26065 (19)	0.13608 (13)	0.0241 (6)
C27	0.8554 (2)	0.38820 (19)	0.07914 (13)	0.0237 (6)
C28	0.6973 (3)	0.51979 (18)	−0.06579 (13)	0.0228 (6)
C29	0.6510 (3)	0.64800 (19)	−0.04945 (13)	0.0242 (6)
C210	0.5397 (3)	0.75956 (19)	−0.11050 (14)	0.0266 (6)
C211	0.4770 (3)	0.7407 (2)	−0.18876 (14)	0.0279 (6)
C212	0.5210 (3)	0.6128 (2)	−0.20462 (14)	0.0277 (6)
C213	0.6311 (3)	0.5021 (2)	−0.14345 (13)	0.0253 (6)
C214	0.4838 (3)	0.89667 (19)	−0.09069 (15)	0.0339 (7)
H21	0.84194	0.32882	−0.02454	0.0406*
H22	1.10678	−0.07693	0.07670	0.0507*
H23	1.20220	−0.07562	0.29049	0.0317*
H24	1.11841	0.11778	0.34719	0.0359*
H25	0.96347	0.32610	0.25221	0.0360*
H27	0.82237	0.46507	0.10348	0.0284*
H29	0.69588	0.65928	0.00377	0.0291*
H211	0.40315	0.81636	−0.23179	0.0335*
H212	0.47534	0.60130	−0.25754	0.0332*
H213	0.66145	0.41471	−0.15429	0.0303*
H214	0.36087	0.91025	−0.06248	0.0507*
H214′	0.48750	0.96918	−0.15007	0.0507*
H214″	0.56659	0.89974	−0.04665	0.0507*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O11	0.0243 (7)	0.0238 (7)	0.0328 (8)	−0.0047 (6)	−0.0006 (6)	0.0013 (6)
O12	0.0235 (8)	0.0288 (8)	0.0377 (9)	−0.0037 (6)	−0.0048 (6)	−0.0024 (7)
N1	0.0209 (9)	0.0205 (9)	0.0246 (9)	−0.0020 (7)	−0.0017 (7)	−0.0050 (7)
C11	0.0259 (11)	0.0175 (10)	0.0269 (11)	−0.0037 (8)	−0.0030 (8)	−0.0077 (8)
C12	0.0228 (10)	0.0201 (10)	0.0335 (12)	−0.0048 (8)	−0.0040 (8)	−0.0094 (9)
C13	0.0282 (11)	0.0197 (10)	0.0308 (12)	0.0002 (8)	−0.0100 (9)	−0.0094 (9)
C14	0.0358 (12)	0.0230 (10)	0.0243 (11)	−0.0020 (9)	−0.0022 (9)	−0.0071 (9)
C15	0.0277 (11)	0.0255 (11)	0.0246 (11)	−0.0026 (9)	0.0016 (8)	−0.0074 (9)
C16	0.0253 (10)	0.0194 (10)	0.0258 (11)	−0.0029 (8)	−0.0030 (8)	−0.0086 (8)
C17	0.0217 (10)	0.0233 (10)	0.0256 (11)	−0.0037 (8)	0.0002 (8)	−0.0103 (9)
C18	0.0242 (10)	0.0180 (10)	0.0231 (10)	−0.0007 (8)	−0.0056 (8)	−0.0072 (8)
C19	0.0282 (11)	0.0211 (10)	0.0213 (10)	−0.0067 (8)	0.0003 (8)	−0.0058 (8)
C110	0.0228 (10)	0.0213 (10)	0.0287 (11)	−0.0058 (8)	−0.0031 (8)	−0.0102 (8)
C111	0.0262 (11)	0.0211 (10)	0.0277 (11)	−0.0039 (8)	−0.0093 (8)	−0.0054 (8)
C112	0.0298 (11)	0.0239 (10)	0.0229 (10)	−0.0093 (9)	−0.0029 (8)	−0.0040 (8)
C113	0.0236 (10)	0.0246 (10)	0.0267 (11)	−0.0064 (8)	−0.0009 (8)	−0.0095 (9)
C114	0.0245 (11)	0.0263 (11)	0.0304 (11)	−0.0042 (9)	−0.0033 (8)	−0.0069 (9)
O21	0.0370 (8)	0.0228 (7)	0.0231 (8)	−0.0030 (6)	−0.0063 (6)	−0.0057 (6)
O22	0.0381 (8)	0.0217 (7)	0.0294 (8)	−0.0007 (6)	−0.0118 (6)	−0.0052 (6)
N2	0.0252 (9)	0.0181 (8)	0.0254 (9)	−0.0032 (7)	−0.0015 (7)	−0.0063 (7)

C21	0.0197 (10)	0.0252 (10)	0.0211 (10)	−0.0067 (8)	0.0012 (8)	−0.0050 (8)
C22	0.0211 (10)	0.0241 (10)	0.0254 (11)	−0.0061 (8)	−0.0025 (8)	−0.0046 (8)
C23	0.0222 (10)	0.0267 (11)	0.0260 (11)	−0.0062 (9)	−0.0046 (8)	0.0000 (9)
C24	0.0293 (11)	0.0378 (12)	0.0223 (11)	−0.0051 (10)	−0.0042 (9)	−0.0096 (9)
C25	0.0297 (11)	0.0310 (11)	0.0302 (12)	−0.0034 (9)	−0.0036 (9)	−0.0128 (9)
C26	0.0204 (10)	0.0245 (10)	0.0271 (11)	−0.0054 (8)	−0.0014 (8)	−0.0070 (9)
C27	0.0211 (10)	0.0228 (10)	0.0276 (11)	−0.0055 (8)	0.0008 (8)	−0.0085 (8)
C28	0.0218 (10)	0.0210 (10)	0.0218 (10)	−0.0060 (8)	0.0040 (8)	−0.0018 (8)
C29	0.0250 (10)	0.0240 (10)	0.0230 (10)	−0.0087 (8)	0.0035 (8)	−0.0051 (8)
C210	0.0226 (10)	0.0220 (10)	0.0304 (12)	−0.0061 (8)	0.0054 (8)	−0.0026 (9)
C211	0.0241 (10)	0.0236 (11)	0.0273 (11)	−0.0043 (8)	0.0012 (8)	0.0030 (9)
C212	0.0278 (11)	0.0301 (11)	0.0238 (11)	−0.0086 (9)	0.0007 (8)	−0.0049 (9)
C213	0.0249 (10)	0.0240 (10)	0.0259 (11)	−0.0065 (8)	0.0036 (8)	−0.0069 (9)
C214	0.0329 (12)	0.0218 (11)	0.0421 (13)	−0.0032 (9)	0.0011 (10)	−0.0063 (9)

Geometric parameters (Å, º)

O11—C11	1.306 (2)	C111—H111	0.9500
O12—C12	1.374 (3)	C112—H112	0.9500
O12—H12	0.91 (2)	C113—H113	0.9500
O21—C21	1.311 (2)	C114—H114	0.9800
O22—C22	1.365 (2)	C114—H114″	0.9800
O22—H22	0.8300	C114—H114′	0.9800
N1—C18	1.421 (3)	C21—C22	1.429 (3)
N1—C17	1.301 (2)	C21—C26	1.423 (3)
N1—H11	1.00 (2)	C22—C23	1.370 (3)
N2—C28	1.420 (3)	C23—C24	1.407 (3)
N2—C27	1.299 (2)	C24—C25	1.369 (3)
N2—H21	0.8700	C25—C26	1.410 (3)
C11—C12	1.428 (3)	C26—C27	1.426 (3)
C11—C16	1.430 (3)	C28—C213	1.396 (3)
C12—C13	1.369 (3)	C28—C29	1.396 (3)
C13—C14	1.413 (3)	C29—C210	1.392 (3)
C14—C15	1.375 (3)	C23—H23	0.9500
C15—C16	1.415 (3)	C24—H24	0.9500
C16—C17	1.430 (3)	C25—H25	0.9500
C18—C19	1.393 (3)	C27—H27	0.9500
C18—C113	1.384 (3)	C29—H29	0.9500
C19—C110	1.393 (3)	C210—C211	1.396 (3)
C110—C114	1.509 (3)	C210—C214	1.507 (3)
C110—C111	1.399 (3)	C211—C212	1.390 (3)
C111—C112	1.388 (3)	C212—C213	1.385 (3)
C112—C113	1.385 (3)	C211—H211	0.9500
C13—H13	0.9500	C212—H212	0.9500
C14—H14	0.9500	C213—H213	0.9500
C15—H15	0.9500	C214—H214′	0.9800
C17—H17	0.9500	C214—H214″	0.9800
C19—H19	0.9500	C214—H214	0.9800

O11···O12	2.719 (2)	C112···H114′^{ix}	2.9100
O11···N1	2.528 (2)	C212···C27^{iv}	3.253 (3)
O11···C114^{i}	3.321 (3)	C113···H114′^{ix}	3.0600
O11···O12^{ii}	2.774 (2)	C214···C22^{vii}	3.538 (3)
O11···O11^{ii}	3.166 (2)	C212···H17^{iv}	3.0800
O12···O11^{ii}	2.774 (2)	C212···H19^{iv}	2.8900
O12···O11	2.719 (2)	C212···H27^{iv}	3.0700
O21···N2	2.550 (2)	C213···H15^{iv}	2.9800
O21···O22^{iii}	2.6965 (19)	C213···H17^{iv}	3.0900
O21···O22	2.7501 (19)	H114′···C111^{ix}	2.9500
O22···O21^{iii}	2.6965 (19)	H114′···C112^{ix}	2.9100
O22···O21	2.7501 (19)	H114′···C113^{ix}	3.0600
O11···H12^{ii}	1.94 (2)	H114″···H23^{xii}	2.6000
O11···H12	2.29 (2)	H214′···H211	2.5400
O11···H11	1.61 (2)	H214′···C13^{xi}	3.0900
O12···H17^{i}	2.8000	H214″···H29	2.3800
O21···H22^{iii}	1.9700	H11···O11	1.61 (2)
O21···H14^{iv}	2.8700	H11···C11	2.22 (2)
O21···H21	1.8200	H11···H113	2.4000
O21···H214^{iv}	2.7500	H12···O11	2.29 (2)
O21···H22	2.3000	H12···O11^{ii}	1.94 (2)
O22···H13^{v}	2.5700	H13···O22^{xiv}	2.5700
N1···O11	2.528 (2)	H14···O21^{iv}	2.8700
N2···O21	2.550 (2)	H15···H213^{iv}	2.5200
C11···C113^{vi}	3.525 (3)	H15···H17	2.4800
C11···C18^{vi}	3.479 (3)	H15···C213^{iv}	2.9800
C12···C111^{vi}	3.500 (3)	H17···O12^{viii}	2.8000
C14···C112^{vi}	3.540 (3)	H17···C19	2.7300
C15···C113^{vi}	3.528 (3)	H17···H15	2.4800
C15···C112^{vi}	3.557 (3)	H17···H19	2.2100
C16···C113^{vi}	3.266 (3)	H17···C213^{iv}	3.0900
C17···C25^{i}	3.581 (3)	H17···C212^{iv}	3.0800
C18···C11^{vi}	3.479 (3)	H19···H17	2.2100
C22···C214^{vii}	3.538 (3)	H19···C17	2.7400
C25···C17^{viii}	3.581 (3)	H19···H212^{iv}	2.3700
C26···C211^{iv}	3.337 (3)	H19···H114	2.3700
C26···C29^{vii}	3.416 (3)	H19···C212^{iv}	2.8900
C27···C212^{iv}	3.253 (3)	H21···O21	1.8200
C27···C211^{iv}	3.258 (3)	H21···C21	2.4200
C28···C28^{iv}	3.464 (3)	H21···H213	2.3200
C28···C29^{iv}	3.595 (3)	H22···O21	2.3000
C29···C26^{vii}	3.416 (3)	H22···O21^{iii}	1.9700
C29···C28^{iv}	3.595 (3)	H23···H114″^{xii}	2.6000
C111···C114^{ix}	3.522 (3)	H23···H113^{ix}	2.5100
C11···H11	2.22 (2)	H25···C16^{viii}	2.7500
C111···C12^{vi}	3.500 (3)	H25···C17^{viii}	2.7400
C112···C15^{vi}	3.557 (3)	H25···H27	2.4400

C112···C14^{vi}	3.540 (3)	H27···C212^{iv}	3.0700
C12···H212^{x}	2.9000	H27···C29	2.7000
C13···H213^{x}	2.9800	H27···H25	2.4400
C13···H214′^{xi}	3.0900	H27···H29	2.1900
C113···C15^{vi}	3.528 (3)	H29···H214″	2.3800
C113···C11^{vi}	3.525 (3)	H29···H27	2.1900
C113···C16^{vi}	3.266 (3)	H29···C27	2.7300
C14···H29^{i}	3.0000	H29···C14^{viii}	3.0000
C114···O11^{viii}	3.321 (3)	H111···C24^{xii}	3.0300
C114···C111^{ix}	3.522 (3)	H111···C23^{xii}	2.9800
C16···H25^{i}	2.7500	H112···H211^{xiii}	2.4600
C17···H25^{i}	2.7400	H112···C23^{ix}	3.0400
C17···H19	2.7400	H113···H23^{ix}	2.5100
C19···H17	2.7300	H113···H11	2.4000
C21···H21	2.4200	H113···C23^{ix}	3.0100
C23···H111^{xii}	2.9800	H114···H19	2.3700
C23···H113^{ix}	3.0100	H211···C112^{xv}	3.0300
C23···H112^{ix}	3.0400	H211···H214′	2.5400
C24···H111^{xii}	3.0300	H211···H112^{xv}	2.4600
C27···H29	2.7300	H212···C12^{x}	2.9000
C29···H27	2.7000	H212···H19^{iv}	2.3700
C111···H114′^{ix}	2.9500	H213···H21	2.3200
C211···C26^{iv}	3.337 (3)	H213···C13^{x}	2.9800
C211···C27^{iv}	3.258 (3)	H213···H15^{iv}	2.5200
C112···H211^{xiii}	3.0300	H214···O21^{iv}	2.7500

C12—O12—H12	112.3 (15)	H114′—C114—H114″	109.00
C22—O22—H22	109.00	C110—C114—H114″	109.00
C17—N1—C18	128.16 (18)	H114″—C114—H114	109.00
C18—N1—H11	124.2 (12)	C110—C114—H114′	109.00
C17—N1—H11	107.7 (12)	O21—C21—C22	119.87 (18)
C27—N2—C28	127.22 (17)	O21—C21—C26	122.64 (17)
C27—N2—H21	116.00	C22—C21—C26	117.48 (17)
C28—N2—H21	117.00	O22—C22—C23	120.09 (18)
O11—C11—C12	119.7 (2)	O22—C22—C21	119.27 (16)
O11—C11—C16	122.90 (19)	C21—C22—C23	120.64 (18)
C12—C11—C16	117.37 (17)	C22—C23—C24	121.03 (19)
O12—C12—C13	120.8 (2)	C23—C24—C25	119.99 (19)
O12—C12—C11	117.88 (17)	C24—C25—C26	120.37 (19)
C11—C12—C13	121.3 (2)	C25—C26—C27	119.97 (18)
C12—C13—C14	120.6 (2)	C21—C26—C27	119.47 (17)
C13—C14—C15	120.22 (19)	C21—C26—C25	120.48 (18)
C14—C15—C16	120.2 (2)	N2—C27—C26	121.76 (18)
C11—C16—C17	118.95 (17)	N2—C28—C29	123.01 (18)
C15—C16—C17	120.7 (2)	N2—C28—C213	116.87 (17)
C11—C16—C15	120.3 (2)	C29—C28—C213	120.11 (19)
N1—C17—C16	120.9 (2)	C28—C29—C210	120.57 (19)
N1—C18—C113	116.4 (2)	C22—C23—H23	119.00

C19—C18—C113	120.49 (19)	C24—C23—H23	119.00
N1—C18—C19	123.11 (17)	C23—C24—H24	120.00
C18—C19—C110	120.52 (18)	C25—C24—H24	120.00
C19—C110—C114	121.28 (18)	C26—C25—H25	120.00
C111—C110—C114	120.28 (19)	C24—C25—H25	120.00
C19—C110—C111	118.5 (2)	C26—C27—H27	119.00
C110—C111—C112	120.7 (2)	N2—C27—H27	119.00
C111—C112—C113	120.30 (19)	C28—C29—H29	120.00
C18—C113—C112	119.5 (2)	C210—C29—H29	120.00
C14—C13—H13	120.00	C29—C210—C211	118.63 (19)
C12—C13—H13	120.00	C211—C210—C214	120.81 (19)
C15—C14—H14	120.00	C29—C210—C214	120.53 (18)
C13—C14—H14	120.00	C210—C211—C212	120.96 (19)
C16—C15—H15	120.00	C211—C212—C213	120.15 (19)
C14—C15—H15	120.00	C28—C213—C212	119.57 (19)
N1—C17—H17	120.00	C210—C211—H211	120.00
C16—C17—H17	120.00	C212—C211—H211	119.00
C110—C19—H19	120.00	C211—C212—H212	120.00
C18—C19—H19	120.00	C213—C212—H212	120.00
C112—C111—H111	120.00	C28—C213—H213	120.00
C110—C111—H111	120.00	C212—C213—H213	120.00
C111—C112—H112	120.00	C210—C214—H214′	109.00
C113—C112—H112	120.00	C210—C214—H214″	109.00
C18—C113—H113	120.00	C210—C214—H214	109.00
C112—C113—H113	120.00	H214′—C214—H214″	109.00
H114′—C114—H114	109.00	H214′—C214—H214	109.00
C110—C114—H114	109.00	H214″—C214—H214	109.00

C18—N1—C17—C16	177.46 (18)	C18—C19—C110—C114	−179.28 (19)
C17—N1—C18—C19	−6.6 (3)	O21—C21—C22—O22	−0.4 (3)
C17—N1—C18—C113	173.7 (2)	O21—C21—C22—C23	179.10 (19)
C27—N2—C28—C213	−166.06 (19)	C26—C21—C22—O22	−179.70 (19)
C28—N2—C27—C26	175.1 (2)	C26—C21—C22—C23	−0.3 (3)
C27—N2—C28—C29	12.5 (3)	O21—C21—C26—C25	−179.88 (19)
O11—C11—C12—O12	−0.2 (3)	O21—C21—C26—C27	3.5 (3)
O11—C11—C12—C13	−178.69 (19)	C22—C21—C26—C25	−0.6 (3)
C16—C11—C12—O12	179.65 (17)	C22—C21—C26—C27	−177.22 (19)
C16—C11—C12—C13	1.1 (3)	O22—C22—C23—C24	−180.0 (2)
C12—C11—C16—C17	−178.94 (18)	C21—C22—C23—C24	0.6 (3)
O11—C11—C16—C15	−179.40 (18)	C22—C23—C24—C25	−0.1 (4)
O11—C11—C16—C17	0.9 (3)	C23—C24—C25—C26	−0.8 (4)
C12—C11—C16—C15	0.8 (3)	C24—C25—C26—C21	1.1 (3)
O12—C12—C13—C14	179.53 (18)	C24—C25—C26—C27	177.7 (2)
C11—C12—C13—C14	−2.0 (3)	C21—C26—C27—N2	−0.6 (3)
C12—C13—C14—C15	0.9 (3)	C25—C26—C27—N2	−177.2 (2)
C13—C14—C15—C16	1.1 (3)	N2—C28—C29—C210	−178.9 (2)
C14—C15—C16—C17	177.86 (19)	C213—C28—C29—C210	−0.4 (3)
C14—C15—C16—C11	−1.9 (3)	C29—C28—C213—C212	0.7 (3)

C15—C16—C17—N1	−176.80 (19)	N2—C28—C213—C212	179.3 (2)
C11—C16—C17—N1	2.9 (3)	C28—C29—C210—C214	177.6 (2)
N1—C18—C19—C110	179.63 (18)	C28—C29—C210—C211	−0.6 (3)
C113—C18—C19—C110	−0.6 (3)	C29—C210—C211—C212	1.4 (3)
C19—C18—C113—C112	0.2 (3)	C214—C210—C211—C212	−176.8 (2)
N1—C18—C113—C112	179.97 (18)	C210—C211—C212—C213	−1.2 (3)
C18—C19—C110—C111	0.5 (3)	C211—C212—C213—C28	0.1 (3)

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) −*x*−1, −*y*+1, −*z*+1; (iii) −*x*+2, −*y*, −*z*; (iv) −*x*+1, −*y*+1, −*z*; (v) *x*+2, *y*−1, *z*; (vi) −*x*, −*y*+1, −*z*+1; (vii) −*x*+2, −*y*+1, −*z*; (viii) *x*+1, *y*, *z*; (ix) −*x*+1, −*y*, −*z*+1; (x) −*x*, −*y*+1, −*z*; (xi) −*x*, −*y*+2, −*z*; (xii) −*x*+2, −*y*, −*z*+1; (xiii) *x*, *y*−1, *z*+1; (xiv) *x*−2, *y*+1, *z*; (xv) *x*, *y*+1, *z*−1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H11···O11	1.00 (2)	1.61 (2)	2.528 (2)	150.8 (18)
O12—H12···O11	0.91 (2)	2.29 (2)	2.719 (2)	108.7 (18)
O12—H12···O11^{ii}	0.91 (2)	1.94 (2)	2.774 (2)	152 (2)
N2—H21···O21	0.8700	1.8200	2.550 (2)	140.00
O22—H22···O21	0.8300	2.3000	2.7501 (19)	114.00
O22—H22···O21^{iii}	0.8300	1.9700	2.6965 (19)	145.00
C13—H13···O22^{xiv}	0.9500	2.5700	3.507 (3)	171.00

Symmetry codes: (ii) −*x*−1, −*y*+1, −*z*+1; (iii) −*x*+2, −*y*, −*z*; (xiv) *x*−2, *y*+1, *z*.